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SHEARING STABILITY OF LUBRICANTS

Y. Shiba

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16. Abstract Shearing stabilities of lubricating oils containing a high mol. wt. polymer as a viscosity index improver were studied by use of ultrasound. The oils were degraded by cavitation and the degradation generally followed first order kinetics with the rate of degradation increasing with the intensity of the ultrasonic irradiation and the cumulative energy applied. The shear stability was mainly affected by the mol. wt. of the polymer additive and could be determined in a short time by mechanical shearing with ultrasound.			
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SHEARING STABILITY OF LUBRICANTS

Y. Shiba

Gentetsudo Gijyutsu Research Laboratory

1. Introduction

Along with reducing the friction and abrasion between surfaces in contact and moving, lubricants also fulfill the simultaneous roles of cooling, cleansing, and sealing. Lubricants which are composed of an appropriately characterized base gasoline to which is added particular additives having stability and necessary functions are commonly used. Because the function of the lubricant must be assured during normal use, the demands of such things as the improvement of the performance of machines and vehicles, conserving the reduction of labor, and the prolongation of the cycle of conservation must be met, and improving the characteristics of the lubricant as well as prolonging its life is important, creating a policy which deals with degradation is a crucial task. /99*

The most fundamental characteristic of lubricants is fluidity. It has become common to use multi-grade oils in which large quantities of high molecular weight polymer additives (such as viscosity index improvers) are added for reduction of labor and reducing energy; the multi-grade oils thus have fluidity with improved thermal properties. However, these lubricants lose efficacy as the additives are damaged by mechanical effects during use, and a decrease in viscosity is observed.

With this background in mind, this report looks at the degradation of lubricants and describes the results of experiments in which primarily ultrasonography was used to evaluate the shearing fracture due to the mechanical action of the high molecular weight polymer additives and shearing stability.

*Numbers in the margin indicate pagination in the foreign text.

2. Degradation of the Lubricants and Dissipation and Degradation of Additives

2.1 Degradation Process of Lubricants

As shown in Table 1, the degradation of lubricants is divided into two major groups, the degradation of the base gasoline in lubricants and the soiling of the lubricant by inclusion of foreign bodies from within the lubrication system or from outside the lubrication system. Furthermore, the dissipation and degradation due to such things as the reactions of the additives are included. The degra-

TABLE 1. Degradation of Lubricants and its Causes

表1 润滑油の劣化とその要因

区分	要因	成分、性状等の変化
潤滑油基油の劣化	酸化劣化 軽質成分の基質 熱 分 解	溶解性および非溶解性生成物 ラッカーカー、スラッジ、カーボン等 粘度増加、酸価増加、不溶解分増加 粘度増加 粘度減少
潤滑油の汚染	潤滑系内部の生成物 摩耗粉、金属、ゴム等 腐食生成物 外部からの異物混入 水、油、酸 水分、燃焼ガス 煙、水、油 空気、酸化物等	不溶解分増加、粘度増加 " " 不溶解分増加、粘度増加 乳化、粘度増加、腐食、変質 引火点低下、粘度減少 比重減化、新油化 " "
添加剤、消耗、劣化	酸化、熱分解 金属表面、酸化 酸性成分の中和 機械的せん断破壊	ラッカーカー 活性成分の減少 塩基価減少 粘度減少

Division	Cause	Changes in Constituents and Characteristics
Degradation of Base Gasoline of Lubricants	Oxidation Degradation	Formation of Soluble or Insoluble Substances (laquer, sludge, carbon, etc) Increased viscosity, Increased acid value, Increase in insoluble fractions

TABLE 1. Degradation of Lubricants and its Causes

Division	Cause	Changes in Constituents and Characteristics
	Volatilization of light weight Components Thermal Decomposition	Increased Viscosity Decreased Viscosity
Soiling of Lubricant	Materials Formed Within the Lubrication System -abrasive particles (metal, gum, etc) -corrosive products Foreign Bodies from Outside the Lubrication System -dust -soot, carbon, ash -moisture, combustible gas -Fuel Oil -Air (Foam)	Increased Insoluble Fraction, Increased Viscosity Increased Insoluble Fraction, Increased Viscosity Emulsification, Increased Viscosity, Corrosion, Tarnish Decreased Ignition Point, Decreased Viscosity Decreased Specific Gravity, Insufficient supply of Fuel
Dissipation and Degradation of Additives	Oxidation, Thermal Decomposition Reaction on Metal Surfaces	Slag Formation Decrease in Active Ingredients

TABLE 1. Degradation of Lubricants and its Causes

Division	Cause	Changes in Constituents and Characteristics
	Neutralization of Acid Components	Decreased Base Value
	Mechanical Shear Fracture	Decreased Viscosity

dation of the base gasoline is thought to be due to fundamental /100 chemical reactions. Many systematic explanations regarding the process of degradation and the products of degradation have been advanced, and a number of experimental procedures for the evaluation of applications have been established. However, because the many complex causes of degradation due to soiling are mutually interrelated, it is usually difficult to make accurate predictions regarding the process of soiling.

However, in the case of operating such things as machines under set conditions, if one assumes that the mixture of soiling substances into the oil and the consumption and supplying of the lubricant can occur in fixed proportions, then the following equation can be used to represent the degradation process due to soiling [2].

$$v_c = \frac{q_c}{q_1} + \exp \left(-\frac{q_1}{V} t \right) \quad (1)$$

In this equation,

v_c : concentration of the products of soiling in the lubricant

q_c : quantity of the products of soiling that are mixed in in unit time

q_1 : rate of consumption of lubricant = rate of supply of lubricant

V : quantity of lubricant

As Figure 1 shows, the concentration of the soiling products increases with time and approaches the threshold value q_c/q_1 . Assuming this type of soiling process, if the concentration of soiling

products is determined when $t = V/q_1$, then one can infer that the threshold value of soiling is q_c/q_1 and it becomes a reference for managing the lubricant.

While the above relationship shows the typical pattern of the degradation process, in actuality it is common for the soiling to progress in an unregulated way, and the effects of the intermittancy of the supply of oil and the effects of the removal of the products of soiling by the lubricating oil filter must be taken into consideration.

2.2 Dissipation of the Additives

Lubricating oil additives, added to lubricating oils because they are endowed with special functional capabilities, can lose the functions owing to reactions caused by chemical activity during use, oxidation, degradation such as thermal dissolution, and consumption. In order to preserve the functions of the additives it is necessary to determine which of the functions are retained and which are lost due to the dissipation of the additive during use.

The dissipation of the additives is investigated using the same assumptions as those used for the process of soiling. If the additives are included in a fixed proportion as components of the lubricating oil which are lost and supplied by reactions in the lubrication system, then the process of the dissipation of the additives is fulfilled by equation (2).

(2)

In this equation: v_a : concentration of additives in lubricating oil

v_o : concentration of additives in the new oil of the lubricating oil

q_a' : dissipation rate of the additive

q_1 : supply rate of the lubricating oil

V : quantity of lubricating oil

Concentration of
Soiling Products: v_c

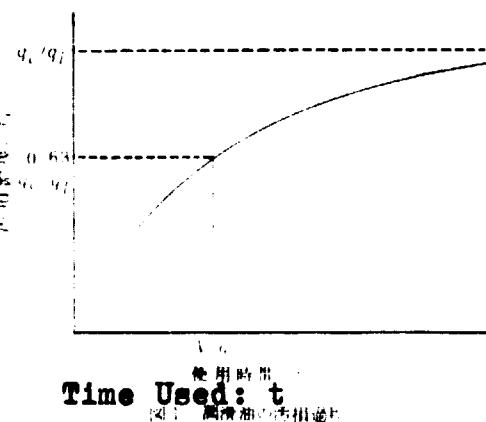


FIGURE 1: Soiling Process of the Lubricating Oil

Concentration of
Additives: v_a

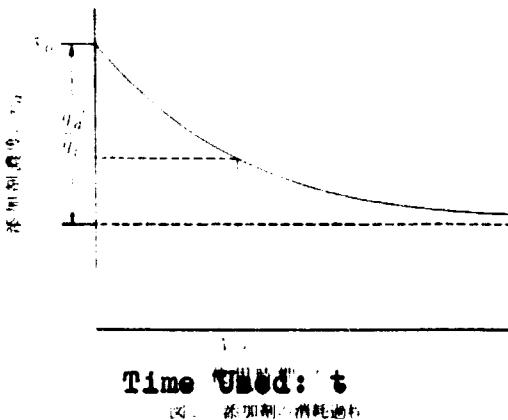


FIGURE 2: Dissipation Process of the Additives

As shown in Figure 2, the concentration of the additives decreased and approached the threshold value determined by the rate of supplying the lubricating oil and the dissipation rate of the additives; the speed of this is dependent on the ratio of the rate of supplying the lubricating oil and the quantity of the oil. Furthermore, when $v_0 < q_a'/q_1$, the threshold value is lost and a fixed concentration of the additive cannot be assured only by supply of new oil.

This way of thinking can be applied to the making of decisions concerning the blending of additives with respect to the basicity (the acid-neutralizing capacity) of engine oils. In order to prevent the corrosion that occurs due to the condensation of the SO_x in the combustion gasses in diesel engines that use combustable oil with a high sulfur content, the basicity of the lubricating oil must be maintained at a fixed level. For this reason one can assume the basicity of the necessary new oil based on equation (2).

In this way, because the important portions of such oils as engine oil and extreme-pressure gear oil which have crucial functions are dependent on the properties of the additives, correctly assessing the description of additives and the residual conditions is desirable /101 for the management of lubrication. However, analyzing the additive with respect to conditions such as basicity and determining the function of it are generally difficult. Infrared absorption spectrography, which is one method used to check the dissipation of additives, has been adapted for determining the functions and components of additives, and it has been suggested that it can be used to manage the state of the lubricating oil from the viewpoint of stability [3].

2.3 Shear Fracture of High Molecular Weight Polymer Additives

The viscosity of lubricating oils generally increases with degradation, however, the viscosity and the index of viscosity of lubricating oils to which high molecular weight polymer additives have been added decrease during use, and the loss of part of the effectiveness of the additive has been observed. Accordingly, in evaluating the state of the lubricating oil to which has been added the high molecular weight polymer additive, in addition to the normal state of fluidity, one must investigate the stability of fluidity under mechanical shearing stress, in other words, the shearing stability.

For many years the phenomenon of the direct change of high molecular weight polymers due to the mechanical action of such things as ball mills has been observed. The intrinsic viscosity of high

molecular weight polymer solutions is also decreased by high-speed agitation; this has been determined to be due to the separation of molecules. Furthermore, this type of shearing fracture has been reported to occur also in conditions of turbidity due to such things as spurtings from small holes and cavitation due to ultrasound and in conditions of laminar flow with high shearing velocity. It has also been suggested in a number of experiments, however there are many points concerning the mechanism of shearing fractures that are still unclear.

3. Shearing Fractures Due to Ultrasonic Irradiation of High Molecular Weight Polymer Additives

3.1 Experiments

With respect to the experimental materials, 6 types of poly-methacrylate additives, representative improvers of the index of viscosity, were used. These materials are shown in Table 2. L-1 to L-4 are polydodecyl-methacrylate compounds dissolved in petroleum type base oils, and N-1 and N-2 are co-polymers of dodecyl-methacrylate and diethylaminoethyl-methacrylate. The polymer concentrations were approximately 40%, and the average molecular weights were determined using the toluene solvent viscosity method.

Ultrasonography was used in the laboratory evaluation of shearing stability. The apparatuses and the methods used in the experiments conformed to ASTM D-2603-70 and Appendix A1, and the conditions under which the experiments were performed are shown in Table 3. The experimental materials which were irradiated for a set interval of time were measured for kinematic viscosity at 25° C, and the variations in the average molecular weight were examined.

3.2 Shearing Fracture Reaction Velocity

The velocity of the reaction is shown by equation (3) if an increase in the velocity, which was proportional to the number of bonds

TABLE 2. Characteristics of the Polymethacrylate Type Viscosity Index Improvers

表2 ポリメタクリレート系粘度指数向上剤の性質

Number 記号	Intrinsic Viscosity 固有粘度	Average Mol. Weight 平均分子量 × 10 ⁵	Average Specific Gravity 平均重合度
L-1*	0.632	2.575	1012
L-2*	0.453	1.633	644
L-3*	0.438	1.560	714
L-4*	0.226	0.631	248
N-1**	0.640	2.624	119
N-2**	0.335	0.833	380

* (CH₂-C(CH₃)-CO₂R)n, R=C₁₂H₂₅

** R=C₁₂H₂₅+(C₁H₈)₂N-C₂H₅

TABLE 3. Conditions of Experimental Irradiation

表3 照射試験条件

Item Experimental Conditions
(Standard)

	実験条件	標準
Frequency	振動数	6 ~ 12 kHz ~ 10 kHz
Output	出力	100 ~ 220 W ~ 200 W
Quantity of Materials	試料容量	20 ~ 50 ml ~ 50 ml
Temperature of Cold Water	冷却水温	5 ~ 45 °C ~ 25 °C
Irradiation Time	照射時間	5 min ~ 3 h

in the molecular chains of the high molecular weight polymer additives which were broken by ultrasonic irradiation, is assumed.

$$\frac{db}{dt} = k (b_0 - b) \quad (3)$$

b : unit volume corresponding to the number of bonds broken

b_0 : number of broken bonds prior to irradiation

Consequently, the velocity constant of the reaction is

$$k = \frac{1}{t} \ln \frac{b_0}{b_0 - b} \quad (4)$$

$$b_0 = cN \left(\frac{1}{M_\infty} - \frac{1}{M_t} \right) \quad (5)$$

$$b = cN \left(\frac{1}{M} - \frac{1}{M_t} \right) \quad (6)$$

In these equations, M , M_0 , and M_∞ are the average molecular weight, respectively, when $t = t$, $t = 0$, and $t = \infty$; c is the concentration of the high molecular weight polymer; and N is Avogadro's number.

Examples of the variation in the kinematic viscosity of the specimens of toluene solvent produced during the experimental irradiation using ultrasound and the results of the calculations, based on this data, of the breaks in bonds due to shearing fractures are shown in Figure 3 and Figure 4. As is clearly shown in Figure 4, the data approximates equation (4), and the shearing fracture reactions in this range approximate first-order reactions.

The results of studying the influence of the concentration of the high molecular weight polymers on the reaction velocity are shown in Figure 5. Other than the large slope of the fracture velocity seen in the initial stage of irradiation under conditions of low concentration, a fairly steady reaction velocity is shown, approximating first order reactions. If this relationship is used to determine the constant of the reaction velocity, as shown in Table 4, the final molecular weights and the velocity constants of almost the same magnitude are obtained. However, the velocity constant

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FIGURE 3. Decreasing Viscosity Due to Ultrasound Irradiation

Kinematic Viscosity (25°C)cSt

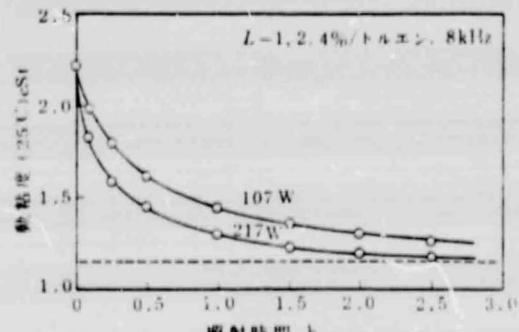


図3 音波照射による粘度低下

Irradiation Time h

FIGURE 4. Breaks in Bonds Due to Shearing Fractures

Number of Bonds
($b_0 - b$) $\times 10^{16}$

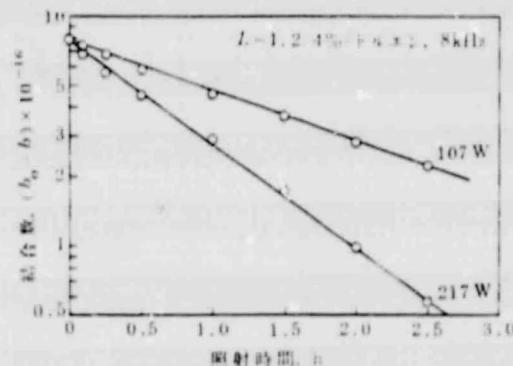


図4せん断破壊による結合の切断

Irradiation Time h

FIGURE 5. Influence of Concentration of High Molecular Weight Polymers on the Velocity of Shearing Fractures

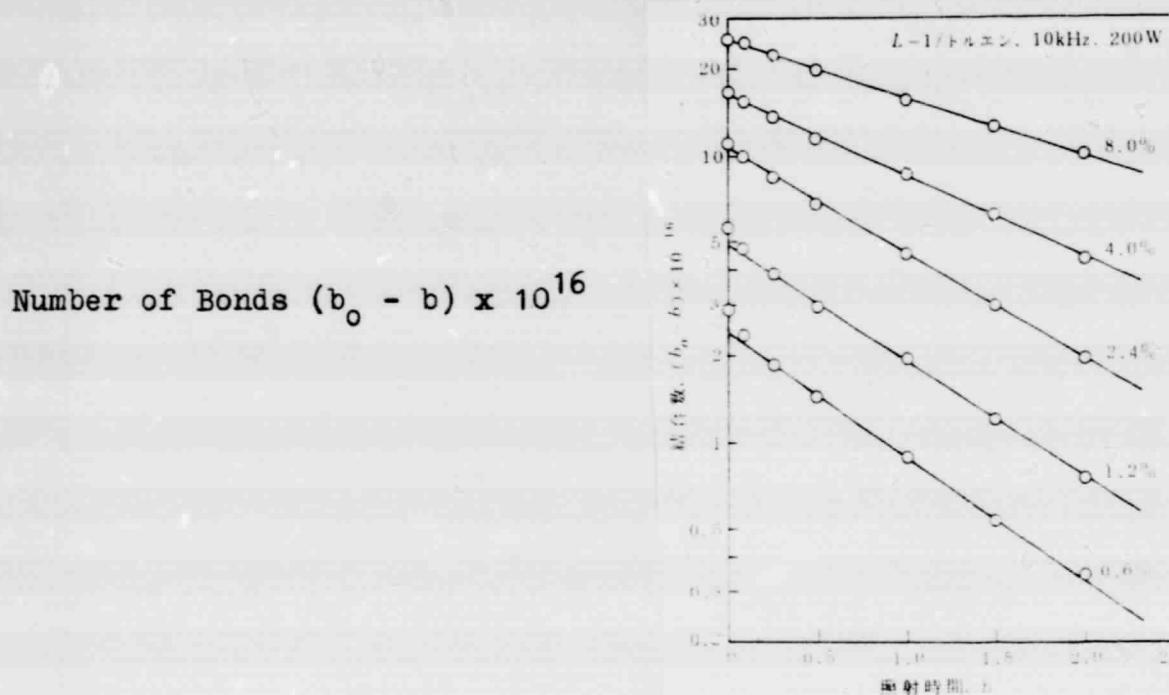


図5 せん断破壊速度に対する高分子濃度の影響

Irradiation Time: h

TABLE 4. Influence of Concentration on the Velocity of Shearing Fractures

Concentration	Initial Concentration	Final Concentration	Final Molecular Weight	Reaction Velocity Constant
濃度 wt %	初期粘度 cSt	最終粘度 cSt	最終分子量 × 10 ⁵	反応速度定数 s ⁻¹ × 10 ⁴
0.6	0.923	0.759	0.84	2.80
1.2	1.269	0.888	0.86	2.51
2.4	2.189	1.202	0.87	2.30
4.0	3.911	1.770	0.92	1.77
8.0	12.04	4.400	1.08	1.26

表4 せん断破壊速度に対する濃度の影響

TABLE 5. Influence of Irradiation Conditions on the Velocity of Shearing Fractures

表5 せん断破壊速度に対する照射条件の影響

Frequency (kHz)	Output (W)	Constant of Reaction Velocity ($s^{-1} \times 10^4$)	Constant of Reaction Velocity/Output ($s^{-1}W^{-1} \times 10^6$)
8	107	1.27	1.18
8	217	2.52	1.16
10	99	1.21	1.22
10	214	2.28	1.07
12	95	1.28	1.35
12	207	2.63	1.28

3.3 Study of the Irradiation Conditions

In general, the ultrasonic irradiation was performed using various instruments under conditions of 6-20 kHz and 100-500 W. The influence of such irradiation conditions as frequency and output on shearing fractures was investigated. In order to clarify problems with the evaluation methods, identical equipment was used and the frequency and output were varied experimentally. Part of the results of the experiments are summarized in Table 5.

Throughout the range of the experimental conditions the constant of reaction velocity was not influenced by the frequency, but it varied in proportion to the output. (Output) x (Time), in other words, if the changes in the kinematic viscosity are graduated with respect to the irradiation energy, then they can be expressed as a single conversion curve as shown in Figure 6. That is to say, under these experimental conditions, the shearing fracture and the velocity constants were determined by the quantity of irradiation energy added to the experimental samples and were not dependent on the frequency. Consequently, it can be thought that the mechanism of shearing fracture is also identical under these conditions.

FIGURE 6. Conversion Curve of Shearing Fracture

Kinematic Viscosity
(25° C), cSt

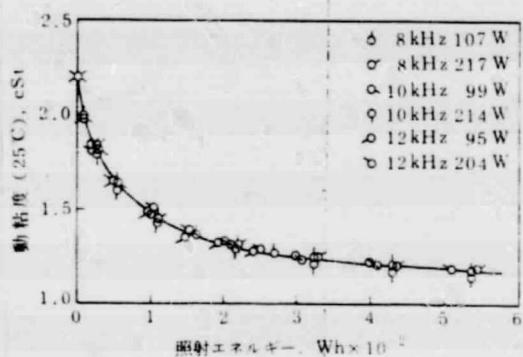


図6 せん断破壊の換算曲線

Irradiation Energy, Wh x 10⁻²

TABLE 6. Shear Stabilized Toluene Solutions of Various Types of
Polymethacrylate Type Additives
(12 kHz, 200 W)

Experimental Sample	Concentration of High Mol. Wt. Polymer (wt%)	Initial Mol. Wt. ($M_0 \times 10^5$)	Final Mol. Wt. ($M \times 10^5$)	Reaction Velocity Constant ($s^{-1} \times 10^4$)
L-1	2.40	2.57	0.87	2.00
L-2	3.00	1.63	0.78	2.17
L-3	2.76	1.56	0.79	1.98
L-4	2.88	0.63	0.51	3.41
N-1	2.40	2.62	0.95	3.28
N-2	2.64	0.83	0.50	3.33

試料	高分子濃度 wt%	初期分子量 $M_0 \times 10^{-5}$	最終分子量 $M \times 10^{-5}$	反応速度定数 $s^{-1} \times 10^4$
L-1	2.40	2.57	0.87	2.00
L-2	3.00	1.63	0.78	2.17
L-3	2.76	1.56	0.79	1.98
L-4	2.88	0.63	0.51	3.41
N-1	2.40	2.62	0.95	3.28
N-2	2.64	0.83	0.50	3.33

表6 各種のポリメタクリレート系添加剤のせん断安定性
トルエン溶液, 12kHz 200W

3.4 Shearing Fractures of Various Types of Additives

The results of ultrasonographic irradiation experiments dealing with various types of polymethacrylate additives which differed in molecular composition and specific gravity are shown in Table 6. When the additives were dissolved to about 6% in toluene and irradiated, although the molecular weight at the end of the irradiation was dependent upon the initial molecular weight, the final molecular weight was slightly less than the initial weight. The improvement in the viscosity index due to the addition of additives was large for the additives with a large initial molecular weight, the variation in the molecular weight due to shearing fracture was large, and shearing stability deteriorated. On the other hand, for L-4 like additives with a small initial molecular weight, the additive effect was inferior and it was necessary to make repeated additions. However, there was only a small decrease in the molecular weight due to shearing, the efficacy of the additive was maintained, and there was a superior shearing stability. The differences due to molecular weight were similarly managed in the additives N-1 and N-2 which differed in composition. Moreover, there were many variations in the constant of the reaction velocity, and it was not thought to contribute much to the stability from a practical standpoint.

3.5 Mechanism of Shearing Fractures

Based on the results of the experiments on the ultrasonic irradiation of high molecular weight polymer additives, the mechanism of shearing fracture can be thought of in the following way.

The decreased viscosity of the solution due to irradiation with ultrasound is not a transient state of dispersion or a change in secondary bonding; it is a permanent loss based on mechanical breaking of the primary chain. This reaction proceeds fundamentally as a first order reaction. It is only dependent upon the total amount

of irradiated energy and it indicates the approach of the final state.

4. Evaluation and Application of Shearing Stability

4.1 Evaluation of Shearing Stability

Practical tests based on the actual running of the machine or the vehicle and laboratory tests are among the methods used to evaluate the shearing stabilities of such things as multigrade engine oil and hydraulic oil which contain high molecular weight polymer improvers of the index of viscosity. In order to make a definitive determination of the final state, the practical tests are essential, and long-term operating tests become necessary. Accordingly, in order to carry out more simple and convenient screening, various laboratory methods have been developed.

Of the laboratory experimental procedures, the nozzle propulsion type shearing stability experimental procedure has been adopted by such places as the JIS, but a weakness of the method is that a long time is required to perform the tests. Among the other experimental procedures, the ultrasonic irradiation method can be considered to be a simple method for evaluating shearing stabilities which requires only small-quantity samples and relatively small increments of time. Therefore this is the most commonly used method now.

It has been shown that the ultrasound irradiation method can be used for lubricating oils containing methacrylate type additives to obtain results relatively consistent with conditions of actual use. The loss of viscosity due to ultrasonic irradiation of engine oil containing methacrylate type viscosity index improvers and two varieties (V. I. = 130-140) of torque converter oil and the results of performing operating tests of the same torque converter oil in a vehicle are shown in Figure 7. According to the results, the loss of viscosity of the engine oil amounted to 20-30%. In contrast to this, the loss for the torque converter oil, parti-

FIGURE 7. Decreased Viscosity of Methacrylate Engine Oil and Torque Converter Oil

Rate of Decrease of Kinematic Viscosity (98.9°C), %

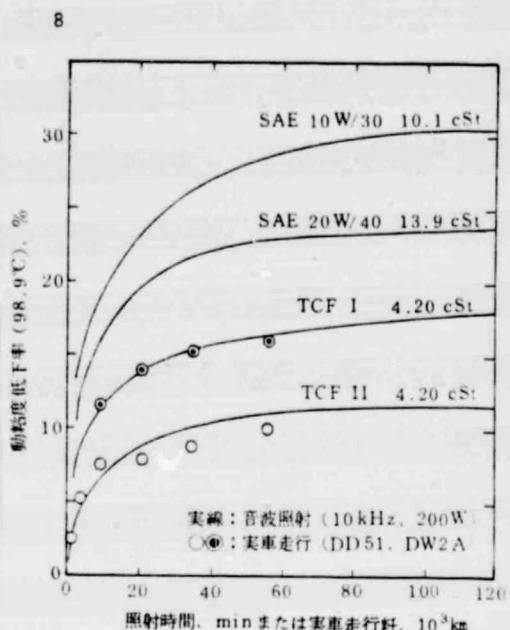


図7 マルチグレード・エンジン油およびトルクコンバータ油の粘度低下

Irradiation Time, Minutes or Kilometers of Vehicle Operation (10³ km)

cularly the T C F - II was about 10%, indicating an excellent shearing stability. Furthermore, if an appropriate length of a cross axle is chosen, the loss of viscosity due to operation of the vehicle can be duplicated by short-term ultrasonic irradiation. Because other degradations posed no real problems in the actual vehicle, the shearing degradation was the only thing that progressed.

However, there are problems with this experimental method which accompany the recent use of various high molecular weight polymers as additives. It has been pointed out that in the case of polyisobutylate type additives the viscosity retention rate in the laboratory experiments is higher than that in the actual vehicles.

Consequently, because the evaluation of lubricating oils containing differing types of additives by means of ultrasonic irradiation is generally not adequate, it is necessary to exercise sufficient care when applying this method. /104

4.2 Application of High Molecular Weight Polymer Additives

Although high molecular weight polymer additives have been developed to improve the fluidity of lubricating oils, because it is possible that during actual use the performance of the oils can be influenced in various ways, beginning with the shearing stability which has not been investigated until now, it is necessary to consider the selection and use of additives from the perspective of their total applicability.

The influence of such things as variations in fluidity due to degradations other than that of shearing stability, non-Newtonian flow, particularly structural viscosity, transient decreases in viscosity such as thixotropy, and increased viscosity due to high pressure forces is a problem with respect to fluidity.

In addition, because large quantities of metals and ashless cleansing agents are added for stabilization of oxidation, cleaning, foaming, and high pressure, careful examination of the compatibility of high molecular weight polymer additives and detergents is necessary.

Accordingly, when high molecular weight polymers are utilized to increase the fluidity, by clearly ascertaining the total capacity of the lubricating oil and the level of fluidity demanded, the most appropriate type, form, and quantity of the additive having a compatibility of fluidity and utility can be selected. Thus, one must consider being able to demonstrate the capacity of the lubricating oil over the entire range of its use.

5. Conclusion

The process of the dissipation of additives and the soiling degradation of lubricating oils during use were investigated with particular attention to the shearing stability of high molecular weight polymer type viscosity index improvers added in order to improve fluidity.

The results of the studies performed with the aid of ultrasonic irradiation can be summarized in the following manner:

- (1) Shearing fractures are based on the mechanical effects of cavitation and can be explained as basically being first order reactions.
- (2) The reaction velocity was proportional to the intensity of the irradiation, and shearing fractures occurred proportionate to the total energy of irradiation.
- (3) The shearing stability of the high molecular weight additives was primarily determined by the molecular weight.
- (4) There were some advantages to evaluating the shearing stability by ultrasonic irradiation due to the short time span required, but it is necessary to exercise caution when applying it to the comparison of various types of additives.

When increasing the performance of lubricating oils by the appropriate application of high molecular weight polymer additives and anticipating the effects of curtailing the consumption of combustable materials and the costs of conservation, it is necessary to consider shearing stability along with adequately ascertaining the effects of the total performance in actual use.

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